Influence of the interaction of light- and self-polymerization on subsurface hardening of a dual-cured core build-up resin composite

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Abstract: OBJECTIVE: To investigate the influence of time delay and duration of photo-activation on subsurface microhardness of a dual-cured resin composite. MATERIAL AND METHODS: A commercially available dual-cured core build-up resin composite (Rebilda DC) was filled in cavities (diameter: 4.0 mm; height: 6.0 mm) of polystyrene molds and light-cured for 20 or 60 s either immediately after the filling procedure (time delay 0 s) or after a time delay of 30, 90, 180 or 300 s. Non-irradiated self-cured specimens served as a control group (n = 15). Specimens were stored in complete darkness and at 100% relative humidity at 37°C for 2 weeks and cross-sectioned. Knoop Hardness Numbers (KHNs) were measured six times per depth and averaged at distances of 0.25, 0.50, 1.00, 2.00, 3.50 and 5.50 mm from the light-exposed surface. Data were statistically analyzed using one- and two-way ANOVA followed by Scheffé's post-hoc test at a level of significance of 0.05. RESULTS: Mean hardness values in all experimental groups ranged between 54.3 ± 2.1 and 58.1 ± 2.3 KHN. Light-curing did not significantly increase composite KHN at any depth measured. Delaying light exposure had no influence on KHN, irrespective of depth. A longer light-exposure time (60 versus 20 s) resulted in significantly higher KHN only at depths of 3.50 and 5.50 mm. CONCLUSION: Photo-activation of the tested dual-cured resin composite provided no clinically relevant benefit compared to self-curing regarding the degree of hardening.

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Influence of the interaction of light- and self-polymerization on subsurface hardening of a dual-cured core build-up resin composite

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Abstract

Objective. To investigate the influence of time delay and duration of photo-activation on subsurface microhardness of a dual-cured resin composite. ***Material and methods.*** A commercially available dual-cured core build-up resin composite (Rebilda DC) was filled in cavities (diameter: 4.0 mm, height: 6.0 mm) of polystyrene molds and light-cured for 20 or 60 s either immediately after the filling procedure (time delay: 0 s) or after a time delay of 30, 90, 180 or 300 s. Non-irradiated self-cured specimens served as control group (n = 15). Specimens were stored completely dark in 100% relative humidity at 37 °C for two weeks and cross-sectioned. Knoop Hardness Numbers (KHN) were measured six times per depth and averaged at 0.25, 0.50, 1.00, 2.00, 3.50 and 5.50 mm distance from the light exposed surface. Data were statistically analyzed using one-way and two-way ANOVA followed by Scheffé's post-hoc test at a level of significance of 0.05. ***Results.*** Mean hardness values of all experimental groups ranged between 54.3 ± 2.1 KHN and 58.1 ± 2.3 KHN. Light-curing did not significantly increase composite KHN at any depth measured. Delay of light exposure had no influence on KHN, irrespective of depth. Longer light exposure time (60 s vs. 20 s) resulted in significantly higher KHN only at 3.50 and 5.50 mm depth. ***Conclusion.*** Photo-activation of the tested dual-cured resin composite provides no clinically relevant benefit compared to self-curing regarding the degree of hardening.

Key words: Dual-cured resin composite, light-polymerization, microhardness

Introduction

Dual-cured resin composites are widely used in modern adhesive restorative dentistry as both core build-up and luting materials. Self-cured or light-cured materials seem appropriate for these applications as well but have limitations. While self-cured materials do not allow the clinician to adjust the setting time individually, light-cured resin composites cannot ensure adequate polymerization in areas with limited access for the curing light. Thus, attenuation of light at increased composite depth leads to potential gradation of the extent of conversion at subsurface levels beyond 2 mm [1]. Even the use of light-transmitting posts cannot compensate for the limited depth of cure, resulting in incomplete polymerization of light-cured resin composites in the apical region of root canals [2,3]. When luting indirect tooth-colored restorations, a combination of scattering, reflection and absorption phenomena reduces light irradiance [4] and, as a result, compromises polymerization of the luting material depending on thickness and shade of the intervening material [5]. Inadequate polymerization has been associated with inferior mechanical properties [6], postoperative sensitivity, microleakage, recurrent caries [7] and pulp irritation caused by residual monomers [8].

Dual-cured resin composites have been developed in an attempt to overcome these limitations, including a redox initiator system in addition to photoinitiators. While superficial areas mainly polymerize through photo-activation, responsible for set-on-command capability and initial stabilization of the restoration, the chemical setting modality is expected to ensure complete polymerization even at deep portions of the material that have received an insufficient intensity of light. However, the incorporation of self- and light-curing modes in the same material has not been shown to guarantee uniform maximal curing. The self-curing option is not only slower [9-11], but also less effective in terms of monomer conversion than when photo-activation is used as a supplement [9,12-15]. Due to incomplete compensation for

deficient light-activation, lower hardness values of dual-cured resin composites have been observed with increased cavity depth [16,17], which may weaken the restoration against occlusal loads. However, studies on curing potential of the two activation modes mainly focused on the initial 24 h of the polymerization reaction. Research into mechanical properties of dual-cured materials after longer observation periods is needed [18].

Despite their independent onset, both curing modes initiate free radical formation and monomer conversion, overlapping each other during the curing process. The exact mechanism of interaction of self- and light-curing is yet unknown. One approach to optimize the extent of polymerization and consequently the structural integrity of the material might be the application of modified light-curing protocols. Duration of light irradiation affects polymerization depth [19], degree of conversion [20] and hardness [21] of resin composites. Recently, it has been speculated that a delay of photo-activation would be beneficial in enhancing the degree of conversion of dual-cured materials as immediate exposure to light could interfere with the self-curing mechanism [22].

Based on these considerations, the purpose of the present *in vitro* study was to systematically investigate the influence of time delay and duration of photo-activation on the extent of polymerization at different depths of a dual-cured core build-up resin composite. Microhardness (as an indirect measure of degree of conversion) was determined two weeks after polymerization initiation in order to assure ultimate double bond conversion and consequently to gain insight into the maximum curing potential of each protocol tested.

Material and methods

Specimen preparation

The dual-curing core build-up resin composite Rebilda DC (VOCO, Cuxhaven, Germany) was used in this study. Details of the tested material are listed in Table I. For fabrication of the composite specimens, molds with through holes (diameter: 4 mm, height: 6 mm) were prepared from white polystyrene. The core build-up material was filled in the cavities of the molds using QuickMix syringes in combination with the corresponding mixing tips type 11 and thin application tips type 4 (VOCO). A 1 mm thick glass plate (Schott, Mainz, Germany) with minimum 90% permeability to light of wavelengths between 380 nm and 2400 nm was placed on each mold squeezing out any excess material. Duration of the filling procedure of each cavity and positioning of the glass plate was measured with a stopwatch and did not exceed 15 s, otherwise the specimens were discarded.

Specimens were light-cured for 20 or 60 s through the glass plate at a standardized distance of 1 mm from the test material either immediately after filling procedure (time delay: 0 s) or after a time delay of 30, 90, 180 or 300 s. Non-irradiated self-cured specimens served as controls. The corresponding experimental groups (n = 15) are described in Table II. Light intensity (High Intensity Mode) of the light-curing unit (Bluephase; Ivoclar Vivadent, Schaan, Liechtenstein) was controlled periodically during the experiment with a radiometer (Optilux, Model 100; SDS Kerr, Danbury, CT, USA) ensuring a constant output intensity of 950 mW/cm^2. Specimens were prepared at ambient room temperature of 28 ± 1 °C. Immediately after their preparation the composite specimens were covered with a light-proof adhesive tape and stored for two weeks in 100% relative humidity at 37 °C.

Determination of Knoop hardness

In order to achieve coplanar centerpieces for determination of microhardness, the molds containing the composite specimens were cross-sectioned with a water-cooled diamond saw (Isomet; Buehler, Lake Bluff, IL, USA) and the cut surfaces were polished with 4000 FEPA P

SiC-paper. Knoop Hardness Numbers (KHN) were determined using a digital microhardness tester (Model No. 1600-6106; Buehler, Lake Bluff, IL, USA). A load of 50 g was applied with a dwell time of 20 s. Hardness measurements were performed at the following distances from the light exposed surface: 0.25, 0.50, 1.00, 2.00, 3.50 and 5.50 mm. For each specimen, six measurements were made at each depth. Over all groups a total of 5'940 hardness measurements were performed.

Statistical analysis

Based on preliminary data with six specimens in each group, a power analysis was performed to determine the number of specimens required in each experimental group. According to this analysis, 15 specimens per group were used to gain power of 94%.

Mean KHN of each depth were calculated from six measurements for each specimen. Data were statistically analyzed using one-way ANOVA for comparison of all groups at the same depth and between different depths within each group. In addition, one-way ANOVA was performed between light-cured groups (groups 2–6 and 7–11) and self-cured control group (group 1) for each depth. Two-way ANOVA was conducted for the factors time delay and light exposure time. Scheffé's post-hoc test was used for multiple comparisons. In all analyses, the level of significance was set at 0.05. Calculations were performed using the SPSS 14.0 software for Windows (SPSS, Chicago, IL, USA).

Results

Mean KHN and standard deviations (SD) of all experimental groups at the respective depths ranged between 54.3 ± 2.1 KHN and 58.1 ± 2.3 KHN as presented in Table III. Comparisons between groups at the same depth revealed that light-curing did not significantly increase

composite KHN at any depth measured ($p > 0.05$). In addition, within each experimental group no significant differences in KHN were detected between different depths ($p > 0.05$). Two-way ANOVA for the factors time delay and light exposure time showed that the delay of light exposure as well as the interaction of the two factors had no significant influence on KHN, irrespective of depth ($p > 0.05$). Figure 1 illustrates the influence of light exposure time on KHN at the respective depths. Longer light exposure time (60 s vs. 20 s) resulted in significantly higher KHN only in the deepest composite layers, at 3.50 mm ($p = 0.038$) and 5.50 mm ($p = 0.018$). However, one-way ANOVA between 20 s light-cured groups (groups 2–6) and controls (group 1) and between 60 s light-cured groups (groups 7–11) and controls (group 1) revealed no significant differences in KHN at any depth measured ($p > 0.05$).

Discussion

Adequate polymerization is a prerequisite for overall clinical success, longevity and biocompatibility of resin composite restorations. Effectiveness of polymerization may be assessed directly or indirectly. Direct methods measuring degree of conversion such as Fourier transform infrared spectroscopy (FTIR) [23] or laser Raman spectroscopy [24] are the most sensitive techniques, but time-consuming and complex. To simplify measurements, various indirect methods have been described in literature. These methods include changes in optical translucency [25], scraping [26], resin leaching [27] and hardness measurements [28]. Knoop hardness has been shown to correlate well with FTIR [27,29,30] and was therefore used in the present study to reflect monomer conversion at different depths of the tested dual-cured resin composite. However, the prediction of an absolute value of degree of conversion by means of an absolute hardness value is not valid, because in addition to the degree of conversion, other factors like filler load, size and type as well as monomer composition and density of network cross-linking affect microhardness of resin composites [31-33].

According to Meredith *et al.* [34], dentin hardness ranges from 50 to 70 KHN, depending on the distance from the amelodentinal junction. Mean hardness values of 56.6 KHN measured for Rebilda DC in the present study therefore predict dentin-like mechanical properties. This observation renders the material suitable for build-ups later prepared for taken up crowns or bridges, since the cutting behavior is similar to dentin. However, microhardness of resin composites may be affected by the molds in which specimens are prepared [35]. White polystyrene was used as mold material due to the fact that its light reflection ranges between that of dentin on the one hand and that of metal on the other hand. This imitates reflection phenomena in cavities that are surrounded by both dental hard tissue and metal matrix commonly used when placing core build-ups.

Temperature is known to have a significant influence on final conversion values of dimethacrylate-based materials by affecting monomer mobility and thus the onset of autodeceleration of the polymerization reaction [36,37]. According to Plasmans *et al.* [38], rubber dam application, recommended for adhesive restorations, results in virtually the same relative humidity and temperature intraorally as in the dental surgery. A preliminary study was performed in order to gain insight into tooth temperature during filling procedure. Tooth 16 of a volunteer was isolated by means of a rubber dam at a constant room temperature of 20 °C. Measurements were taken 15 min after application of the rubber dam using thermocouple (TES-1303; TES, Taipei, Taiwan) and revealed a tooth surface temperature of 28 °C. This observation is consistent with results published by Pogrel *et al.* [39] who found that tooth temperature after rubber dam application varies from 27.5 to 29.2 °C. Therefore, composite specimens in the present study were prepared at ambient room temperature of 28 ± 1 °C prior to storage for two weeks in 100% relative humidity at 37 °C.

During hardness measurements light had to be applied to the specimens. In a second preliminary study it was therefore evaluated whether exposure to light after two weeks of dark storage influences microhardness of the resin composite. For that purpose, four specimens of

Rebilda DC were allowed to self-cure for two weeks, whereas two of the specimens were subsequently light irradiated for 120 s with the light-curing unit in High Intensity Mode. No differences in hardness values at the top surface were determined between light irradiated and non-irradiated groups, indicating that the restricted polymer network developed during two weeks of self-cure did not allow any additional mobility of the polymer chains, which justified light application during hardness determination.

The present study demonstrated that light irradiation does not affect microhardness of the tested resin composite (Table III, Figure 1), indicating similar extent of polymerization in self- and dual-curing mode. This finding suggests a different trend to that in previous reports in which photo-activation of dual-cured materials resulted in higher degree of conversion compared with chemical activation alone [9,12-15]. El-Mowafy *et al.* [7] found that for three of eight examined dual-cured resin cements, self-curing produced hardness values less than 50% of those obtained when dual-curing was used. They concluded that the self-curing option is not appropriate to achieve sufficient hardening, even after one week of storage. However, it has also been reported that polymerization behavior of dual-cured resin composites is strongly material-related and can vary as a function of composition [40]. Therefore, conclusions on the curing mechanism of a specific composite material may not be transferred to other products. According to Hasegawa *et al.* [41], dual-cured materials differ markedly in relative contents of light and chemically activated catalysts. Differences in degree of conversion among materials when subjected to various curing protocols might consequently be attributed to variations in catalyst systems. As in the present investigation similar hardness values were observed irrespective of composite depth (Table III), it might be inferred that the tested material exhibits high levels of chemical curing activator compensating for attenuation of light energy in the deep part of the restoration. The equal degree of polymerization within the core material may support a uniform distribution of stress along tooth-material interfaces under load. Even though the results of the present study may be limited to the specific resin

composite tested, the evaluation of only a single core build-up material is justified due to the large number of measurements that were performed in order to gain insight into the exact polymerization mechanism in different subsurface composite layers. This research project particularly intended to thoroughly investigate the influence of the moment of photo-activation on the interaction of the self- and light-curing mode, a parameter occasionally overlooked in studies on polymerization behavior of dual-cured materials. Rebilda DC was chosen as test material since the exact quantitative composition of both base and catalyst paste was revealed by the manufacturer. In general, such information is not available for commercially products, but only for model materials.

A previous study directly measured C=C conversion of Rebilda DC at 15 min postmix and found that monomer conversion was significantly lower in self-curing mode compared with dual-curing [12]. The similar extent of polymerization of the same material in the presence or absence of light two weeks after polymerization initiation, as observed in our study, indicates an increased impact of the self-curing mode with time and may be explained by the difference in velocity between the two curing modes. Lee *et al.* [10] reported up to about 320 times slower curing speeds by chemically induced cure than by additional light irradiation, suggesting that very early measurements may severely underestimate the progressive hardening potential of the self-curing mode. Microhardness is generally determined 24 h after polymerization initiation. However, a substantial increase in degree of cure following the first day postmix has previously been described for dual-cured resin cements subjected to self-curing only [42]. Consequently, setting times in the present study were extended to two weeks, in order to assure ultimate double bond conversion and hence to allow an assessment of the maximum hardening potential of the various polymerization scenarios tested.

Recently, Meng *et al.* [43] observed that light irradiation shortly after composite application inhibits the self-curing mechanism of dual-cured materials, probably by

entrapping polymerization promoters and unreacted monomers in the polymer network. In order to avoid premature interaction of the two curing modes, it has been recommended to delay photo-activation of dual-cured resin composites to the maximum time clinically possible [22]. The present study revealed that the moment of photo-activation does not affect microhardness of the tested core build-up material. It is therefore supposed that immediate photo-activation, despite causing a rapid increase in viscosity of the polymer matrix, does not hinder migration of activated free radicals responsible for further chemically induced polymerization. Moreover, delayed photo-activation seems not to cause synergic effects of the curing modes given that neither short (30 s) nor medium (90 s, 180 s) or long (300 s) delay periods were able to increase the extent of polymerization. Thus, the structure of the polymer network at the start of light-polymerization does not influence the curing potential of the tested material. In accordance with our results, Moraes *et al.* [13] reported that delayed photo-activation does not affect the degree of conversion of dual-cured materials. On the other hand, recent literature demonstrates that delaying light irradiation of dual-cured resin composites reduces microleakage in Class II restorations [44]. Therefore, delayed photo-activation procedures, allowing for some initial conversion by the self-curing mode, might be beneficial in reducing shrinkage stress of dual-cured materials. This speculation warrants investigation in future studies.

Evaluation of duration of photo-activation revealed that longer exposure time (60 s vs. 20 s) results in significantly higher hardness values in the deepest composite layers, at 3.50 and 5.50 mm (Figure 1). Although statistically significant, differences in microhardness between groups light-cured for 20 or 60 s respectively were less than 2% in both depths and therefore extremely small in absolute terms. Thus, the effect of longer photo-activation seems not to be clinically relevant for the tested material.

The self-curing mechanism of dual-cured resin composites is usually based on a redox reaction of benzoyl peroxide with aromatic tertiary amines, generating free radicals that will

break the aliphatic carbon double bonds to start the polymerization process. Both, peroxides and amines are organic compounds with limited storage stability. Dentists should be aware that deterioration of these components may compromise the effectiveness of the self-curing mechanism even before the expiration date, particularly at increased storage temperature. For the present study, fresh material was provided by the manufacturer, which was stored cool when not in use. These conditions may have contributed to the good performance of the chemically induced setting reaction.

Based on the results of the present *in vitro* study, it can be concluded that photo-activation of the tested core build-up resin composite provides no clinically relevant benefit compared to self-curing regarding the degree of hardening achieved two weeks after polymerization initiation. The dominant self-curing mode allows application of the tested material in areas that are inaccessible to the curing light. Further research on the effect of different curing protocols on the extent of polymerization is warranted with other dual-cured core build-up resin composites.

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Figure legends

Figure 1. Mean (SD) KHN of non-irradiated controls (group 1) and of all groups light-cured (LC) for 20 s (groups 2–6) or 60 s (groups 7–11) at the respective depths of the tested material Rebilda DC (n = 15).

Table 1: Manufacturer's information about the tested composite material Rebilda DC.

Composition (wt%)	Filler size (μm)	Shade	Batch #	Manufacturer
Base: bariumborosilicate glass (63.6), fumed silica (6.0), Bis-GMA (5.0), UDMA (19.0), DDDMA (5.0), CQ (0.2), DABE (0.4), N,N-Bis (0.6), BHT (0.2) *Catalyst:* bariumborosilicate glass (64.4), fumed silica (6.0), Bis-GMA (5.0), UDMA (19.0), DDDMA (5.0), BPO (0.4), BHT (0.2)	1.5	white	0806461	VOCO, Cuxhaven, Germany

Bis-GMA: bisphenol-A-glycidyldimethacrylate; UDMA: urethane dimethacrylate; DDDMA: dodecanediol dimethacrylate; CQ: camphorquinone; DABE: dimethylaminoethylbenzoate; N,N-Bis: N,N-Bis-hydroxyethyl-p-toluidine; BHT: butylated hydroxytoluene; BPO: benzoyl peroxide.

Table II. Experimental groups.

Group	Time delay (s)	Light exposure time (s)
1 (control)	-	0
2	0	20
3	30	20
4	90	20
5	180	20
6	300	20
7	0	60
8	30	60
9	90	60
10	180	60
11	300	60

Table III. Mean (SD) KHN of experimental groups at the respective depths of the tested material Rebilda DC (n = 15).

Group	TD	LET	KHN					
			0.25[a]	0.50[a]	1.00[a]	2.00[a]	3.50[a]	5.50[a]
1 (control)	-	0 s	55.8 (2.3)	56.0 (2.5)	57.2 (2.6)	56.8 (2.3)	56.6 (2.2)	54.9 (2.5)
2	0 s	20 s	55.1 (2.6)	55.8 (2.8)	55.7 (2.5)	56.8 (2.4)	56.4 (1.8)	55.9 (2.4)
3	30 s	20 s	56.5 (2.4)	56.2 (1.9)	56.8 (2.5)	57.1 (2.4)	57.3 (1.9)	57.2 (2.7)
4	90 s	20 s	56.6 (2.7)	57.1 (2.2)	58.1 (2.3)	57.8 (2.3)	57.5 (2.3)	56.0 (1.9)
5	180 s	20 s	56.5 (2.7)	56.5 (2.7)	56.5 (1.7)	56.5 (2.6)	56.4 (3.0)	54.3 (2.1)
6	300 s	20 s	55.1 (2.4)	55.8 (2.0)	57.0 (2.1)	57.6 (2.3)	57.0 (2.0)	55.4 (2.7)
7	0 s	60 s	56.5 (2.7)	55.9 (2.4)	56.6 (2.2)	57.5 (2.5)	57.9 (2.1)	57.1 (3.0)
8	30 s	60 s	56.2 (3.1)	56.6 (3.3)	57.1 (2.9)	56.9 (2.8)	57.4 (2.1)	57.0 (1.9)
9	90 s	60 s	56.1 (3.8)	56.3 (3.5)	56.9 (2.3)	57.7 (2.7)	57.7 (2.3)	56.3 (2.6)
10	180 s	60 s	55.1 (1.7)	55.6 (1.6)	56.2 (1.6)	57.3 (3.8)	57.8 (3.1)	56.6 (3.1)
11	300 s	60 s	55.7 (3.7)	56.4 (3.2)	56.8 (3.7)	56.7 (1.9)	57.7 (2.4)	56.6 (1.9)

[a] Depth (mm); TD: time delay before light exposure; LET: light exposure time.

None of the differences are statistically significant at the 0.05 level.

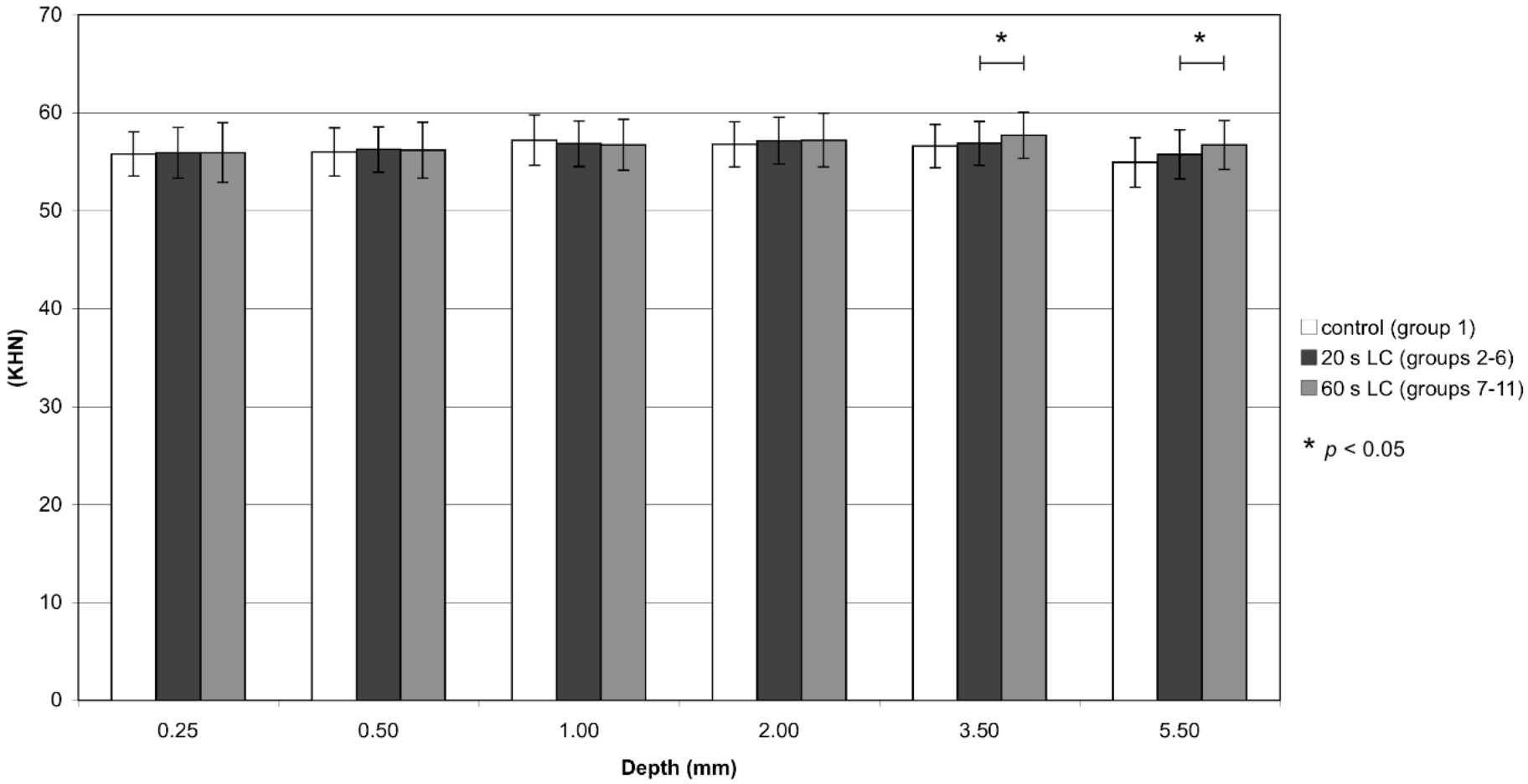
70
60
50
40
30
20
10
0
(KHN)
*
*
0.25
0.50
1.00
2.00
3.50
5.50
Depth (mm)
control (group 1)
20 s LC (groups 2-6)
60 s LC (groups 7-11)
* p < 0.05

Figure 1